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Floating Zone Process for Drawing Small Diameter Fibers of Refractory Materials

Composites of different metals or of metals and ceramics permit the creation of materials that take advantage of the desirable properties of the constituents and minimize their undesirable properties. Superior properties can be achieved in fiber-reinforced composite products, and it is believed that these products can be produced economically by mass production techniques. One of the more promising groups of such materials under investigation today comprises metallic composites composed of a metal matrix reinforced with refractory fibers such as the ceramic aluminum oxide, Al_2O_3 .

Since the development of such composites is contingent upon the development of high-temperature-resistant refractory fibers, it is necessary to develop processes for the production of fibers which exhibit high strength and negligible creep at temperatures of $1093^\circ C$ ($2000^\circ F$) and above. Processes producing continuous fibers are highly desirable from the point of view of both manufacture and application.

A process for drawing continuous fibers of ceramic materials has recently been developed and has potential for adaptation to mass production. The process produces controlled purity, very high strength, single crystal fibers of materials with melting points to $4000^\circ C$ ($7232^\circ F$). It has been used to make single crystal fibers of highly refractory ceramics such as Al_2O_3 , TiC and Y_2O_3 .

The fibers are produced directly from the melted ceramic without the use of a crucible, thus eliminating a principal source of contamination encountered in other processes. The heating source for this process is a 400 watt CO_2 laser, which is capable of producing temperatures of $4000^\circ C$ ($7232^\circ F$).

To form a single crystal ceramic fiber, the laser is focused on the tip of a feed rod of the desired material (such as Al_2O_3 , plain or doped), and power is increased until the tip of the feed rod begins to melt. A single crystal seed of the same material is brought into contact with the molten tip of the feed rod in such a manner as

to initiate single crystal growth onto the seed from the molten drop. The seed crystal is then drawn away at a rate predetermined to achieve the desired dimensions (diameters between 0.025 cm (0.010 inch) and 0.065 cm (0.025 inch)) for the solidified single crystal filament. Since the feed rod is cylindrical and the surface tension forces tend to produce symmetrical low energy shapes, the solidified filament is also cylindrical with a glass-smooth surface that has very few defects.

A Cr_2O_3 -doped Al_2O_3 fiber which was produced by this process is a macroscopic oxide fiber with the highest strength at elevated temperature that has ever been produced. Room temperature strengths obtained to date are 3.93×10^9 N/m² (570,000 psi) in tension and 9.66×10^9 N/m² (1,400,000 psi) in buckling. Average elevated temperature tensile values are 1.12240×10^9 N/m² (162,666 psi) at $1093^\circ C$ ($2000^\circ F$) and 0.75×10^9 N/m² (109,000 psi) at $1316^\circ C$ ($2400^\circ F$). Stress rupture results at $1093^\circ C$ ($2000^\circ F$) and $1316^\circ C$ ($2400^\circ F$) are 1,524,000 cm (600,000 inches) for 100 hours and 1,219,200 cm (480,000 inches) for 100 hours, respectively. This doped Al_2O_3 fiber is twice as strong as any other macroscopic fiber for which data are available, on the basis of stress rupture-density at $1093^\circ C$ ($2000^\circ F$) and $1316^\circ C$ ($2400^\circ F$); it is thermodynamically more stable than high-temperature, high-strength, low-density fibers such as carbon.

This process has also been used to produce single crystal fibers which can be made by no other known process. Small diameter fibers of Y_2O_3 and TiC , which melt at $2410^\circ C$ ($4370^\circ F$) and $3200^\circ C$ ($5702^\circ F$), respectively, have been produced.

Notes:

1. This process is being used to produce ceramic fibers for both metal matrix and ceramic matrix fiber-reinforced composites for applications at operating temperatures above $1093^\circ C$ ($2000^\circ F$).

(continued overleaf)

2. The small diameter fibers produced by this process can also be utilized for making high-temperature light pipes, and for making jewel bearings by slicing the fibers into disks. For these applications, this process should offer a reduction in cost over conventional processes by eliminating the need to machine bulk rods of Al_2O_3 down to small diameter fiber size.
3. The following documentation may be obtained from:
National Technical Information Service
Springfield, Virginia 22151
Single document price \$3.00
(or microfiche \$0.95)

Reference: NASA CR-72811 (N71-19222), Production of Oxide Fibers by a Floating Zone Fiber Drawing Technique

4. Technical questions may be directed to:
Technology Utilization Officer
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Reference: B72-10491

Patent status:

Title to this invention has been waived under the provisions of the National Aeronautics and Space Act /42 U.S.C. 2457(f)/, to Arthur D. Little, Inc., Cambridge, Massachusetts 02140.

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